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STRUCTURE OF ELECTRODEPOSITED CHROMIUM ON GUN STEEL(U)

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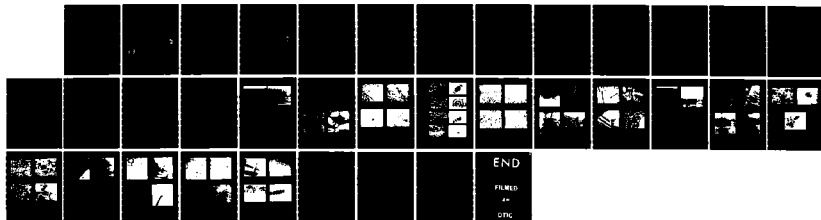
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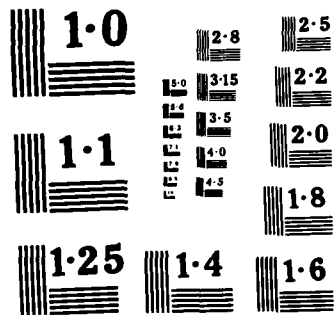
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TECHNICAL REPORT ARLCB-TR-85036

# STRUCTURE OF ELECTRODEPOSITED CHROMIUM ON GUN STEEL

M. H. KAMDAR

R. M. FISHER

SEPTEMBER 1985

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT CENTER  
LARGE CALIBER WEAPON SYSTEMS LABORATORY  
BENET WEAPONS LABORATORY  
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Scanning and transmission electron microscope examination of electrodeposited chromium has revealed that plating conditions affect the size and orientation of the very fine individual crystallites. Plating at 55°C results in a bright, very hard, 1180 Knoop-hardness number (KHN), coating comprised of 0.1 µm diameter grains with a strong <111> fiber texture oriented perpendicular to the surface of the base metal. Plating at higher temperatures such as 85°C, or (CONT'D ON REVERSE)		



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## INTRODUCTION

Previous detailed studies using a variety of analytical techniques have shown that the "white layer" which forms on the bore surface of cannon tubes during firing is a consequence of intense carburization of the interior surface by the high temperature and high carburizing activity of the gas produced from the explosive charge (refs 1-3). This results in the formation of high carbon molten steel, which is a very corrosive medium and can cause very rapid deterioration of the interior surface, making it necessary to replace the cannon tube.

A possible means of alleviating the extent of white layer formation is to protect the surface by applying a thin coating, perhaps up to 100  $\mu\text{m}$  in thickness, of chromium by electrodeposition (ref 4). A long-standing difficulty with such thin coatings is the occurrence of extremely high tensile stresses in the plane of the deposit (refs 5-12). These usually lead to cracking, either during the plating operation itself or during subsequent heating.

The magnitude of the tensile stresses and the extent of the contraction that subsequently occurs, depends on the plating conditions, particularly current density and electrolyte temperature. Samples of 4340 type gun steel were electroplated with chromium and used for metallographic examination by light and electron microscopy.

The metallography of electroplated chromium is well-known for being difficult. The grain size is generally extremely small, internal stresses are large due to high concentrations of defects, the presence of cracks can cause

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References are listed at the end of this report.

difficulty with polishing or thinning, and etching to reveal the micro-structure is not easy because of the very etch-resistant nature of chromium.

#### EXPERIMENTAL PROCEDURES

Four samples of chromium plated steel, as described in Table I, were examined. One, designated as sample A, is representative of typical electroplating conditions which result in a rather hard "bright" deposit with a very high degree of contraction. Other samples electroplated under conditions which produce lower hardness and much less contraction and cracking were examined, along with sections of a chromium plated cannon tube after test firing.

TABLE I. SAMPLES OF CHROME PLATING EXAMINED BY ELECTRON MICROSCOPY\*

Sample	Preparation	Hardness	Grain Structure
A Hard Chrome Hc 12-80-1	30 A/dm <sup>2</sup> 55°C	1150 KHN (190 after annealing at 900°C)	0.1 μm dia. strong fiber texture <111>
B L.C. chrome LC-12-80-3	120 A/dm <sup>2</sup> 85°C	650 KHN (188 after annealing at 870°C)	1.5 μm weak texture
C L.C. chrome HEX. 10-80-nt	120 A/dm <sup>2</sup> well-aged bath	610 KHN	1.8 μm weak texture
D Fired cannon 8312-09-001	-	-	0.1 μm strong texture

Samples were mounted and polished in cross-section and in planar section in order to observe the crack structures in both orientations. The

\*These specimens were supplied by Dr. E. Chen, Research Branch, Benet Weapons Laboratory.

metallographic appearance of cracks in both light and electron microscopes was found to depend on preparation conditions, as discussed in the following sections of this report.

Thin foils were prepared by ion milling for transmission microscopy in the USS million-volt electron microscope. Slices cut parallel to the surface were ground to about 50  $\mu\text{m}$  in thickness. Because of the cracks, only small pieces survived this process and truly satisfactory thin foils could not be obtained in all cases.

#### METALLOGRAPHIC OBSERVATIONS

Optical micrographs of cross and surface sections of sample A of bright hard chromium are shown in Figure 1. Electrolytic polishing-etching reveals the presence of numerous cracks perpendicular to the plated surface, as shown in Figure 1b, and on the planar section shown in Figure 1c. At higher magnification in the scanning electron microscope, cracks were revealed very clearly in both cross and parallel sections of the chrome plate, as shown in Figure 2. The appearance of the cracks suggested that they contain some unknown material, although this effect was not fully reproducible between the usual alternate polishing and etching cycles.

The results of very deep electroetching of the chrome plate are shown in Figure 2. It may be seen that a thin film of what is presumed to be amorphous chromium oxide forms at the cracks (ref 13). This structure will be discussed further in a section dealing with the nature of microcracks in electro-deposited chromium.

Transmission electron micrographs of a thin foil of sample A are shown in Figure 3. The pattern of microcracks and the extremely fine grain size are illustrated at several different magnifications. The electron diffraction pattern from a selected area is also included in the figure. Indexing of the diffraction rings reveals that the (200) reflections are completely missing. This is indicative of an extremely pronounced  $\langle 111 \rangle$  fiber texture. The apparent grain size, as determined by dark field micrographs, as in Figure 3d, is about  $0.1 \mu\text{m}$ . This grain size is about five times larger than typical values given in the literature which were based on x-ray line-broadening measurements. The probable reason for the difference is that the grains themselves contain a very high dislocation density in a rather ill-defined substructure. The high dislocation density of about  $10^{14}$  would markedly affect line broadening and indicate an apparent finer grain size than is actually present.

When the specimen is tilted so that  $\langle 111 \rangle$  axes of the columnar grains are not parallel to the beam in the high-voltage microscope, a rather characteristic electron diffraction pattern appears, as illustrated in Figure 4. In this sequence of images and diffraction patterns, the foil was tilted about the axis of the crack running across the center of the field of view. The total range of tilting was 45 degrees, that is from +15 degrees to -30 degrees, as indicated on the figure. Gaps which occur in the diffraction rings and the characteristic striped pattern result from an oblique view of a fiber-textured specimen. Some micrographs of another tilt series are shown in the dark field in Figure 5. One of the striking features of the microcracks is that they are discontinuous and "ligaments" of metal connect some grains

all through the specimen. Presumably these connections are the reason that the foil, although extremely fragile, remains intact and can be mounted and examined in the high-voltage microscope. This crack structure will be discussed further in the following section.

The crack pattern in the electroplated chromium becomes more pronounced after heating for one hour at 900°C and air cooling. Examples of cross and parallel sections are shown in Figure 6. It does not appear that the cracks are any more numerous than before but have become wider, presumably because of further contraction during annealing of the electrodeposit. The hardness decreased to 160 KHN as a result of the annealing treatment. This is a larger drop than might be expected from the grain size change, indicating that part of the high hardness results from the dense dislocation substructure.

High voltage electron micrographs after annealing sample A for one hour at 900°C are shown in Figure 7. These show that although recrystallization has occurred, many of the ligaments along the cracks remain. It is more likely that they are chromium oxides formed from the codeposition of anhydrous chromium oxide in the grain boundary cavities during plating (ref 14).

Sample B (chromium electrodeposited at 85°C) was examined in the manner described in the preceding section of this report. Optical micrographs of the cross and surface sections are shown in Figure 8, and scanning electron micrographs in Figure 9. It may be noted that the hardness of 650 KHN is about half that of sample A and the crack spacing is about ten times coarser. High voltage electron microscopy of ion-thin sections of the 85°C sample revealed a much coarser grain size and more random grain orientations, as illustrated in Figure 10.

The grain structure of the chromium in sample C, which was electro-deposited at 85°C with a used plating solution, was found to be generally similar to that observed in sample B. High voltage electron micrographs of specimens examined as-plated and after annealing are shown in Figure 11. Annealing resulted in little grain coarsening but did produce more equiaxed grains and eliminated much of the dislocation substructure.

Scanning and high voltage electron microscopy were also used to examine the chromium electroplate on a sample of fired cannon. Micrographs of the cross-section and surface are shown in Figures 12 and 13 after mechanical polishing, and in Figure 14 after electropolishing. The grain, dislocation, and crack structures in thin foils are illustrated in the high voltage micrographs in Figure 15. The cracks are wider and more complex after firing as compared with annealing at 870°C to 900°C for laboratory samples. During firing the surface is heated to >1100°C for a few milliseconds and is also exposed to explosive pressures. A more detailed study of the crack structures should be carried out.

#### RESULTS AND DISCUSSION

The results of optical, scanning, and high voltage electron microscopy of samples of chromium electrodeposited under various conditions have revealed the following salient features:

1. The grains are small, 0.1  $\mu\text{m}$  in diameter, and highly oriented ( $\langle 111 \rangle$  fiber texture) when electrodeposition occurs at 55°C. At 85°C or when old plating solution is used, the grains are much larger (1.0 to 1.5  $\mu\text{m}$ ) and more randomly oriented.

2. The cracking tendency decreases with increasing grain size.
3. The cracks which form along grain boundaries are discontinuous and remain so after annealing.
4. The structure of the cracks is rather complex and frequently they occur as very closely-spaced double cracks.
5. An amorphous film forms at the crack during electropolishing.

It appears from this study that the high tensile stresses present in electrodeposited chromium result from the fine grain structure, and the tendency for cracking should therefore be controllable. A theoretical model for the stresses is under consideration and will be reported in the future. It suggests lower stresses in thin deposits. The cracks in sample A are spaced about 10 to 20  $\mu\text{m}$  apart and are about 0.2  $\mu\text{m}$  in width, which corresponds to a relaxation of tensile strain of about one to two percent. The crack spacing observed here corresponds to 100 to 200 grain diameters, indicating an average void space of 10° to 20°A per grain boundary. This amount is not unreasonable for the unequilibrated grain structure produced during electrodeposition. Although the stresses in chromium electroplate are generally found to be tensile, "compressive" stresses have been reported in some cases. Presumably such apparent compressive stress is a result of cracking during plating and the deposition of chromium within the cracks during further plating.

The lower hardness obtained by plating at high temperature, or by annealing previously, results from both a decrease in dislocation density and grain coarsening. In terms of a hardness grain-size "Petch plot", the contributions of these two effects are about equal. It is known that one to

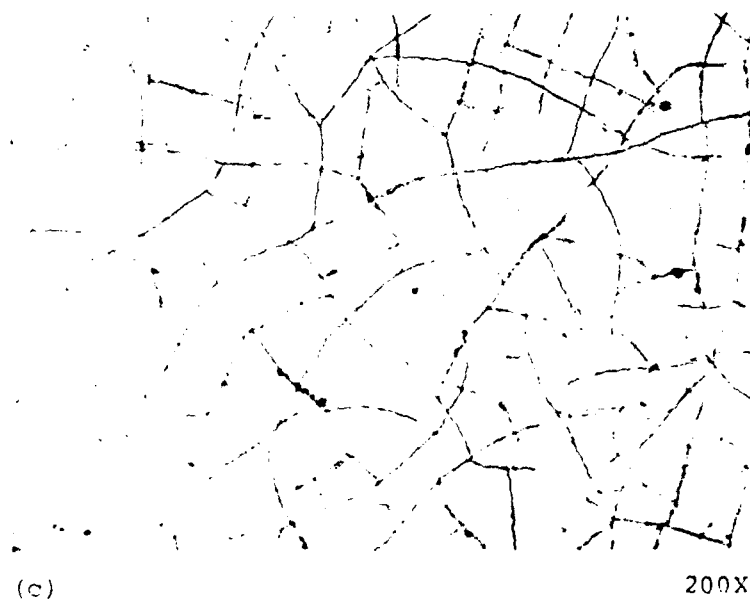
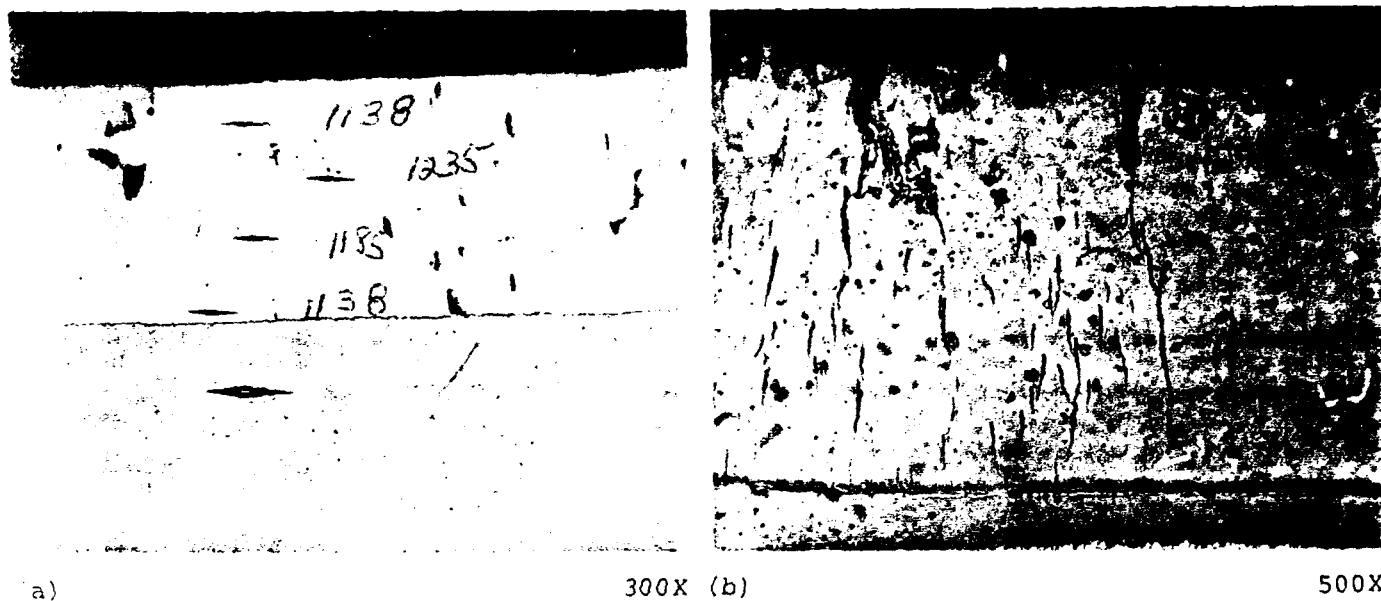
two percent of  $\text{Cr}(\text{OH})$  is trapped within the deposited film, and this effect apparently results in the formation of chromium oxide ( $\text{Cr}_3\text{O}_4$ ). Some weak electron diffraction evidence for this phase was obtained in the high voltage electron microscope.



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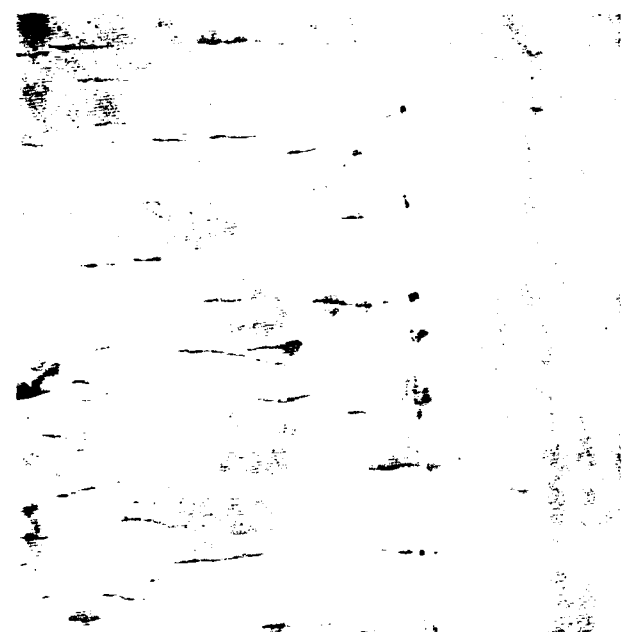
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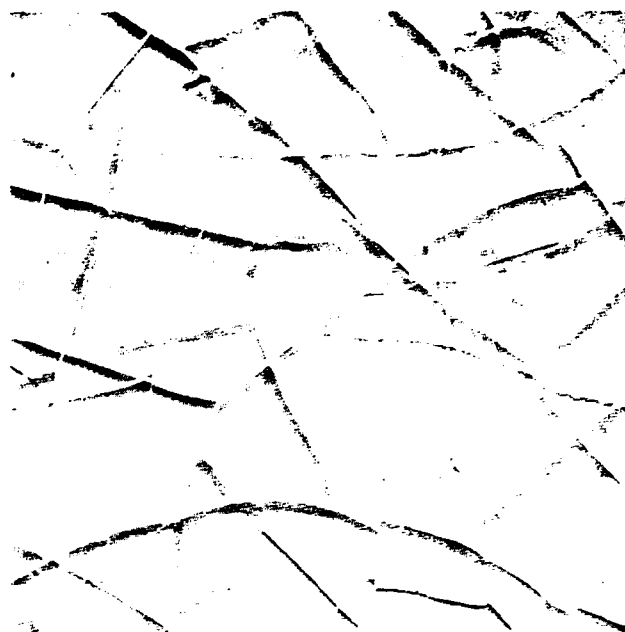
Optical micrographs of chromium electrodeposited at 55°C showing Knoop hardness values on cross section (a), and microcracks revealed by electropolishing cross section (b), and surface section (c).

Figure 1



(a)

1000X



(b)

500X



(c)

200X

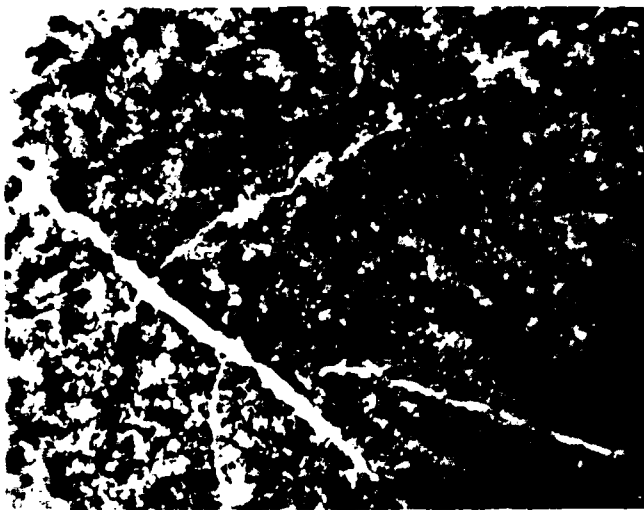


(d)

1000X

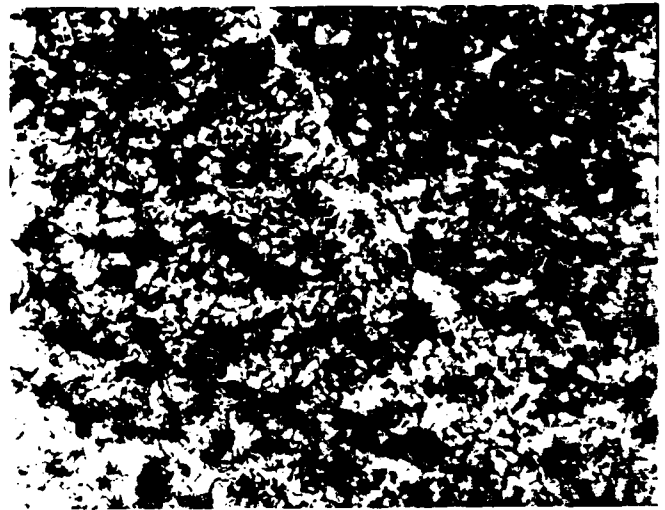
Scanning electron micrographs of chromium electrodeposited at 55°C and electropolished to reveal microcracks; cross section (a), and surface (b), (c), (d). Prolonged electropolishing enhances formation of amorphous film (c), (d).

Figure 2



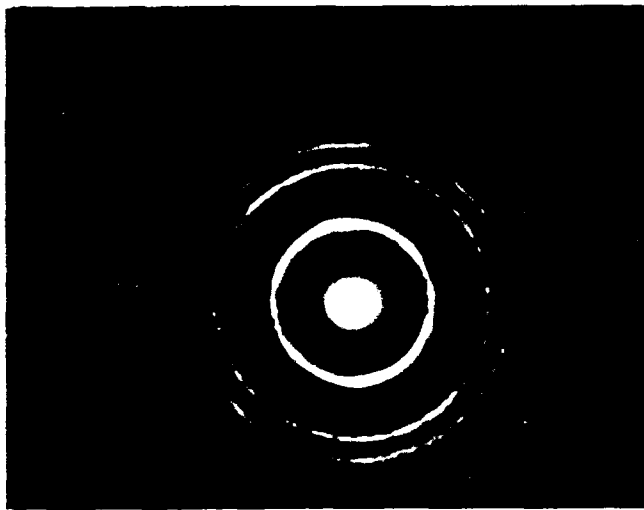
(a)

11,000X



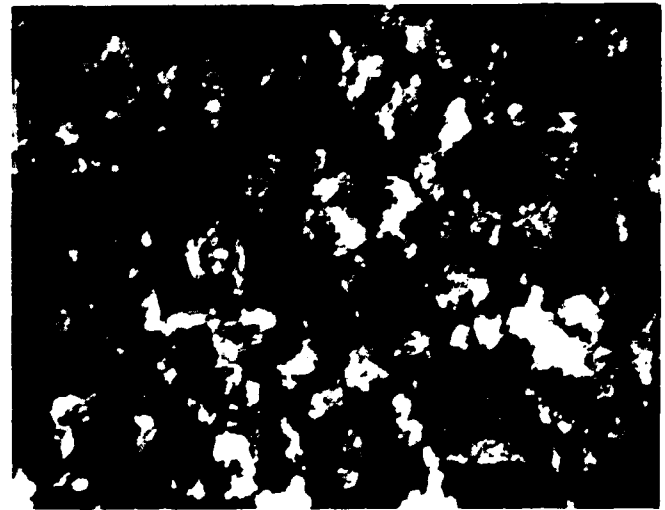
(b)

15,000X



(c)

SAD



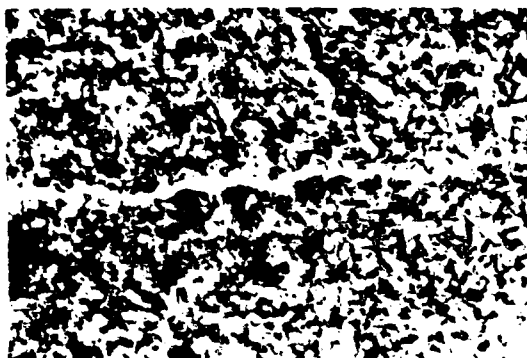
(d)

60,000X

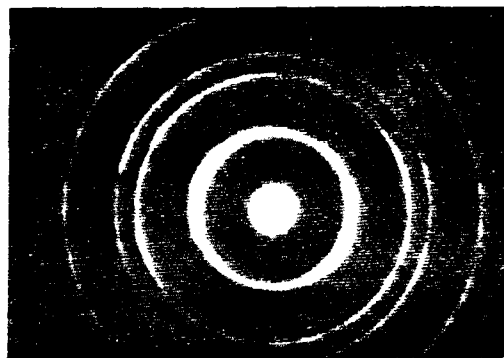
High voltage transmission electron micrographs of chromium electrodeposited at 55°C showing microcracks (a), fine 0.1 $\mu$ m grain size (b), electron diffraction pattern (c), and dark field image (d).

Figure 3

+15°

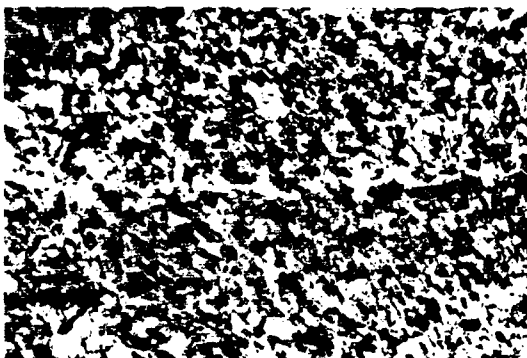


0°

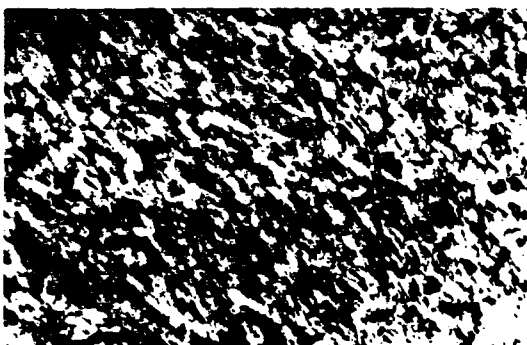


15,000X

-15°



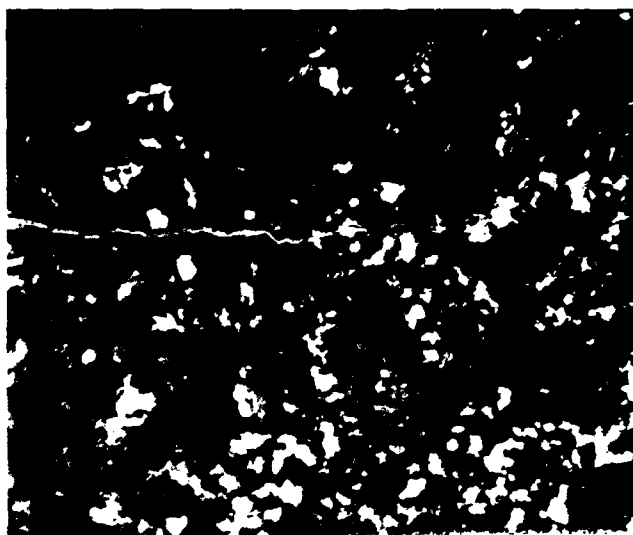
-30°



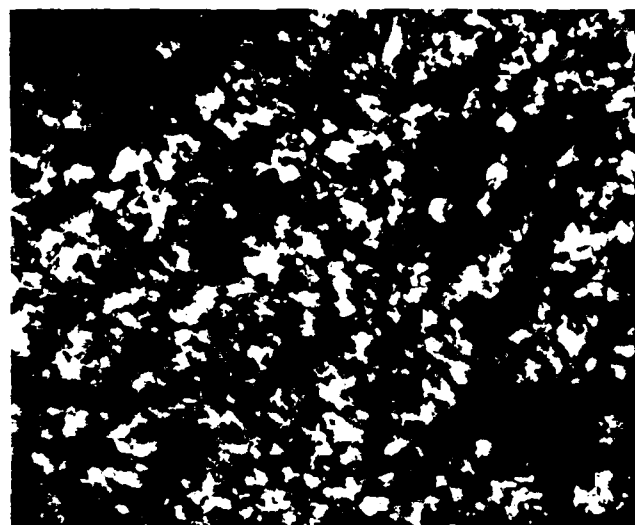
High voltage transmission electron micrographs of chromium electrodeposited at 55°C. Sample was tilted around an axis along the microcrack. Missing 200 reflection and discontinuous arcs are characteristic of a 110 fiber texture parallel to the film thickness direction.



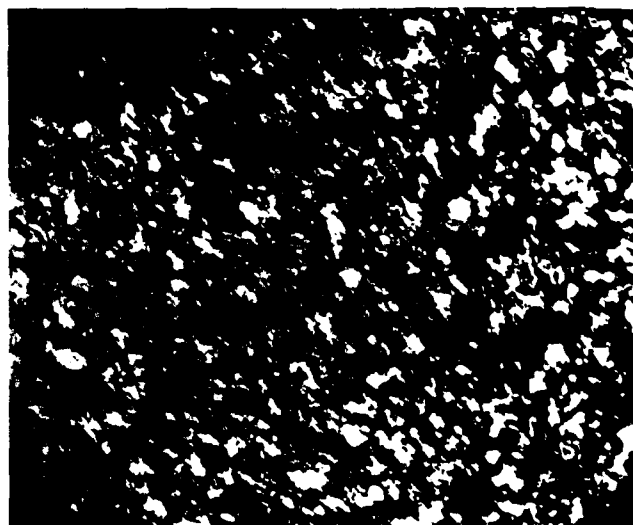
(a)  $+15^\circ$  15,000X



(b)  $0^\circ$  15,000X



(c)  $-15^\circ$  15,000X



(d)  $-30^\circ$  15,000X

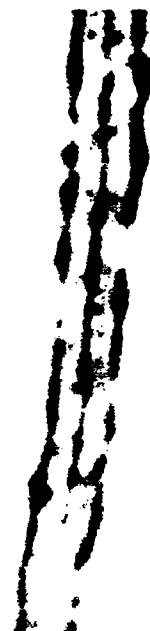
High voltage transmission electron micrographs (all dark field) showing grain clusters with similar orientations.

Figure 5



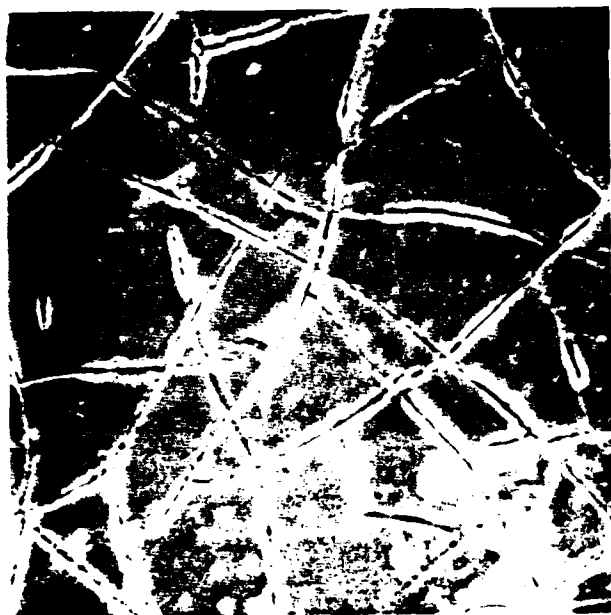
(a)

1000X



(b)

10,000X



(c)

1200X



(d)

6000X

Scanning electron micrographs of chromium electrodeposited at 55°C and then annealed at 900°C for 1 hr to enlarge microcracks; both cross sections (a), (b), and surface sections (c), (d) were electropolished.





(a)

2000X



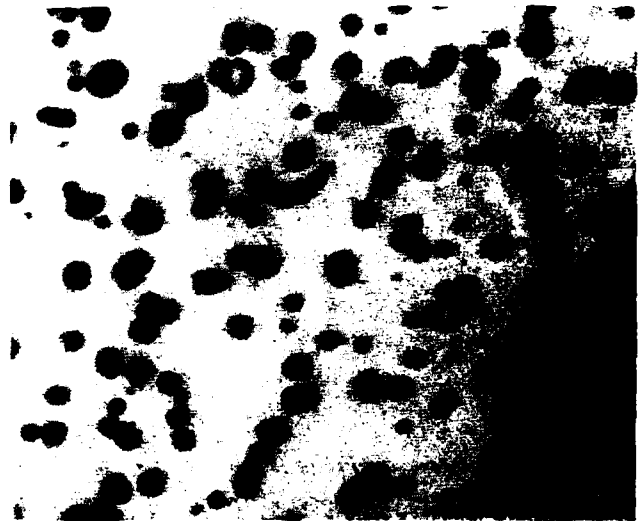
(b)

10,000X



(c)

30,000X

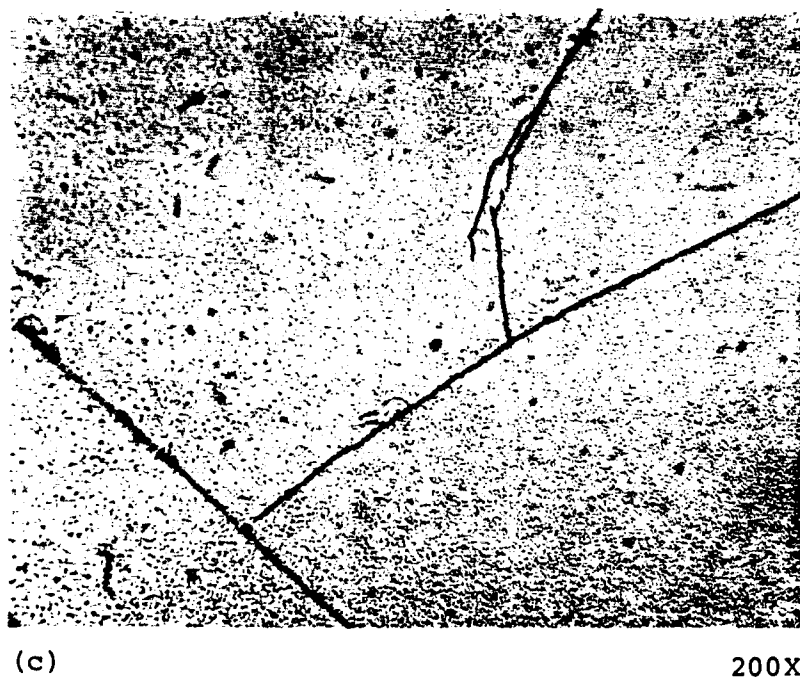
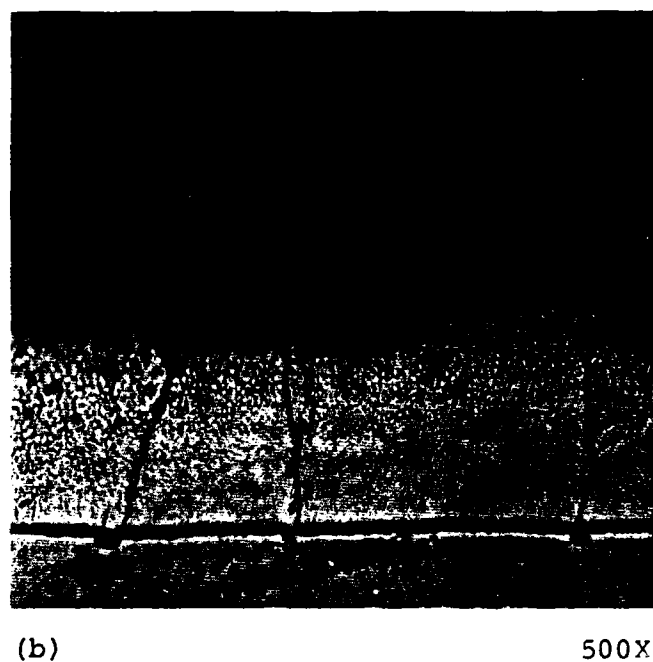
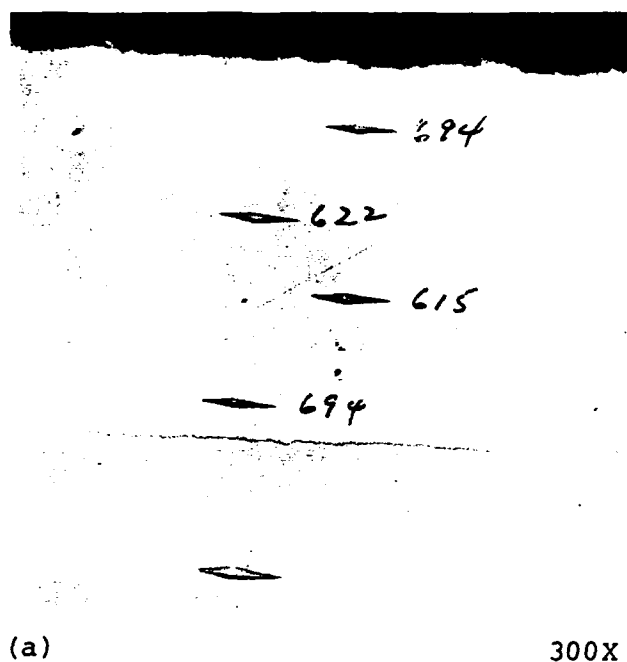


(d)

120,000X

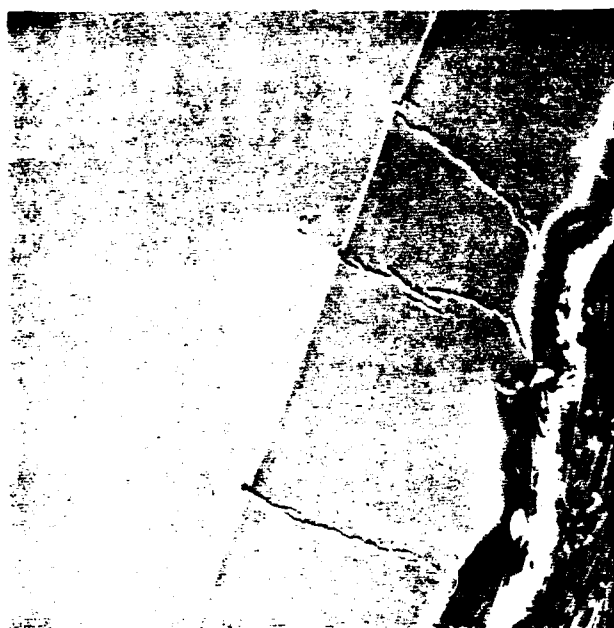
HVEM transmission micrographs showing chromium electro-deposited at 55°C and then annealed for 1 hr at 900°C resulting in crack coarsening (a), recrystallization (b), (c), and formation of fine precipitates or vacancy loops (d).

Figure 7



Optical micrographs of chromium electrodeposited at 85°C showing Knoop hardness values (a), and coarse microcracks in cross section (b), and surface section(c).

Figure 8



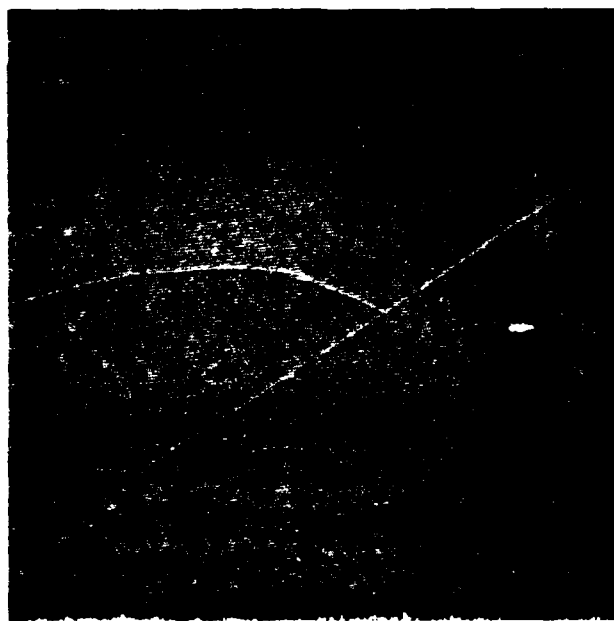
(a)

500X



(b)

5000X



(c)

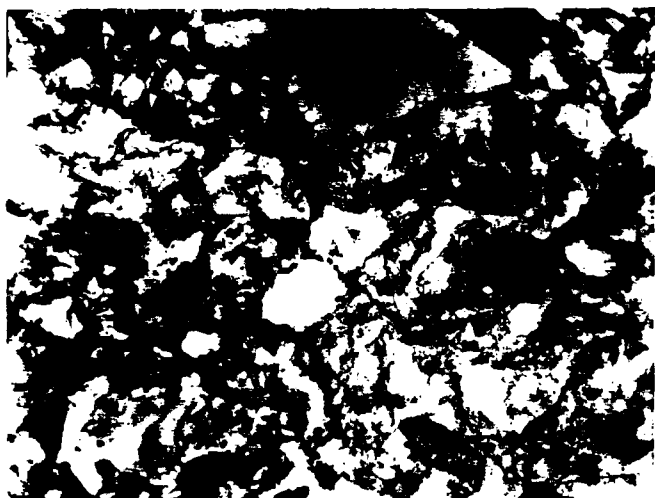
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(d)

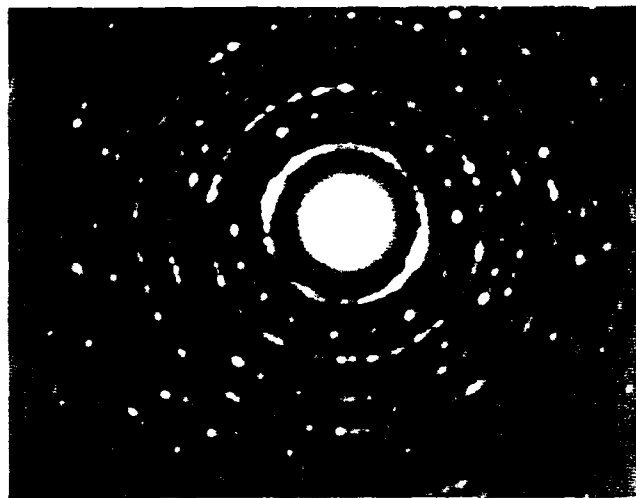
300X

Scanning electron micrographs of chromium electrodeposited at 85°C showing coarse microcrack structure; cross sections (a), (b); surface sections (c), (d).



(a)

15,000X



(b)

SAD

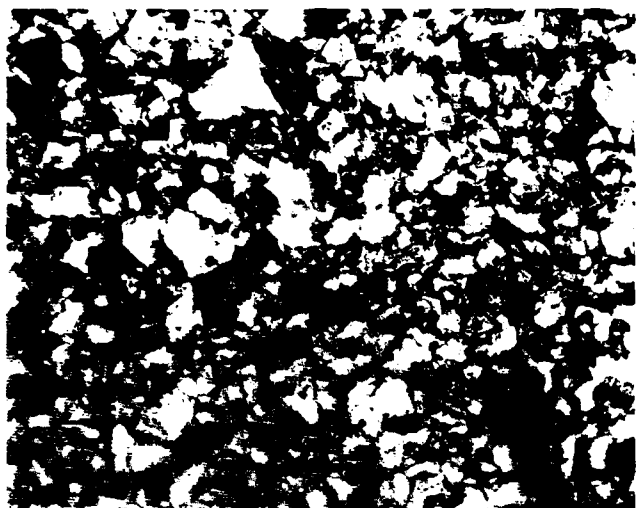


(c) Dark Field

15,000X

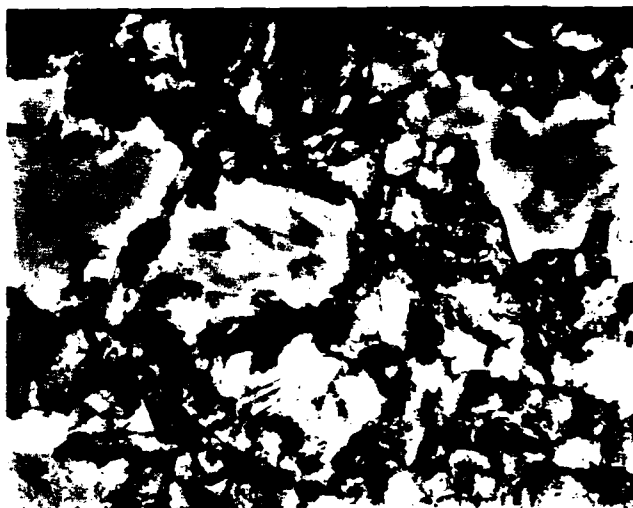
HVEM micrographs of chromium electrodeposited at 85°C showing dense substructure in the 1μm grains which exhibit only a weak fiber texture.

Figure 10



(a)

5000X



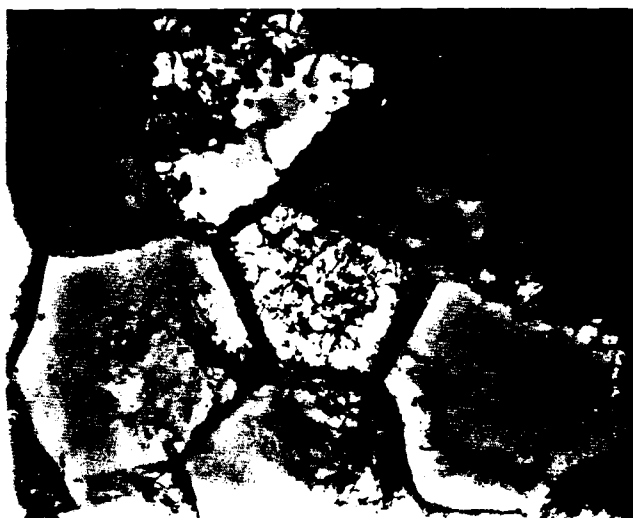
(b)

15,000X



(c)

7500X

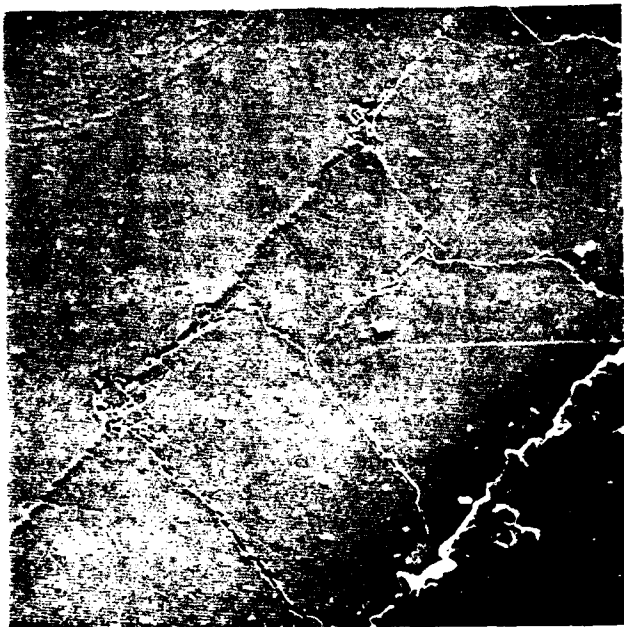


(d)

15,000X

High voltage electron micrographs of chromium electrodeposited at 55°C with used plating solution; as deposited (a), (b), and after annealing 1 hr at 870°C (c), (d).

Figure 11



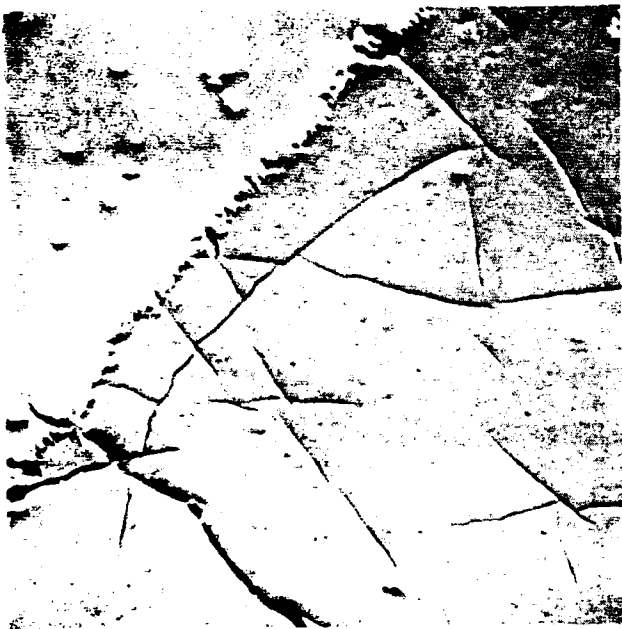
(a)

200X



(b)

500X



(c)

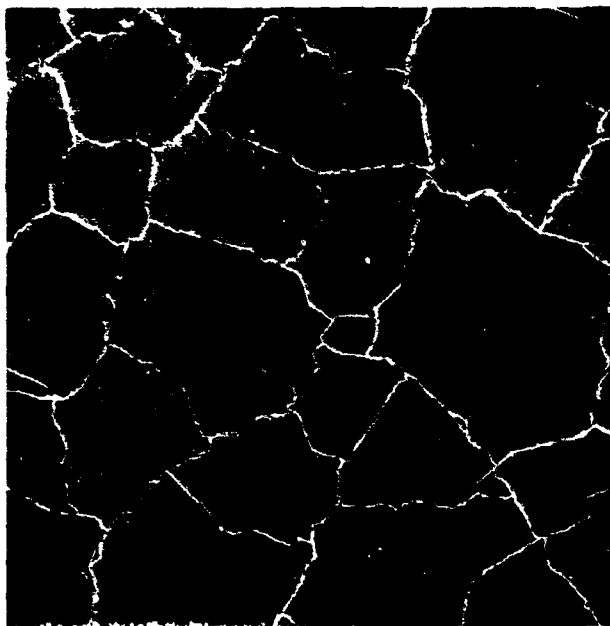
1000X



(d)

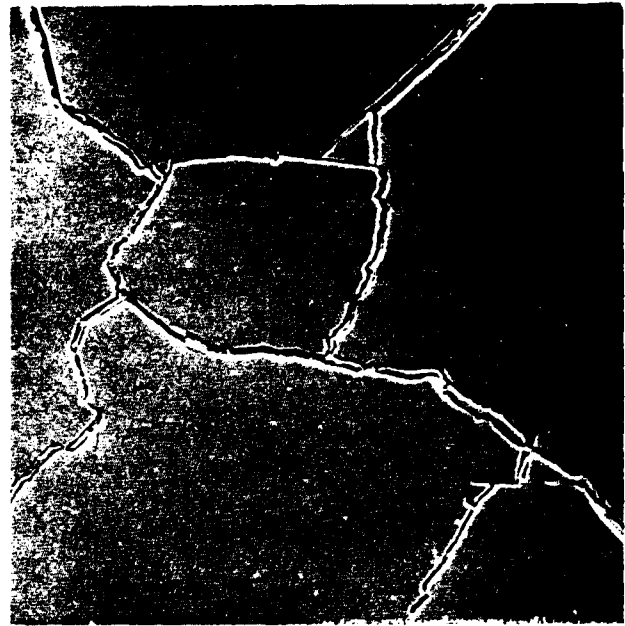
5000X

Scanning electron micrographs of mechanically polished cross section of chromium electroplate on a fired cannon.



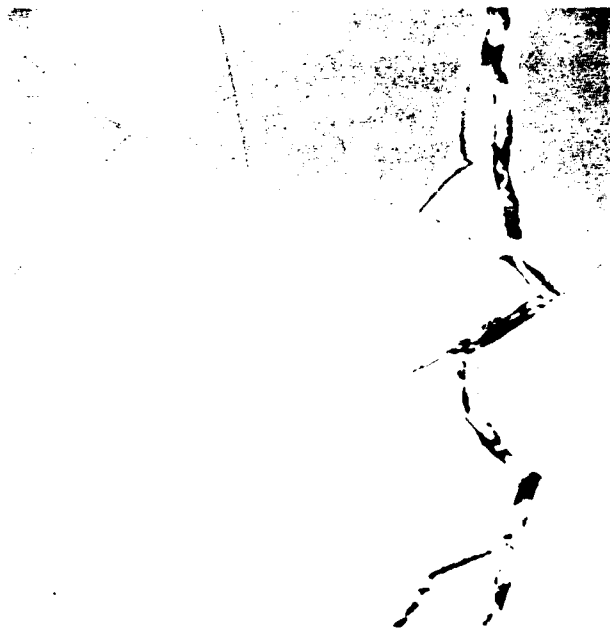
(a)

100X



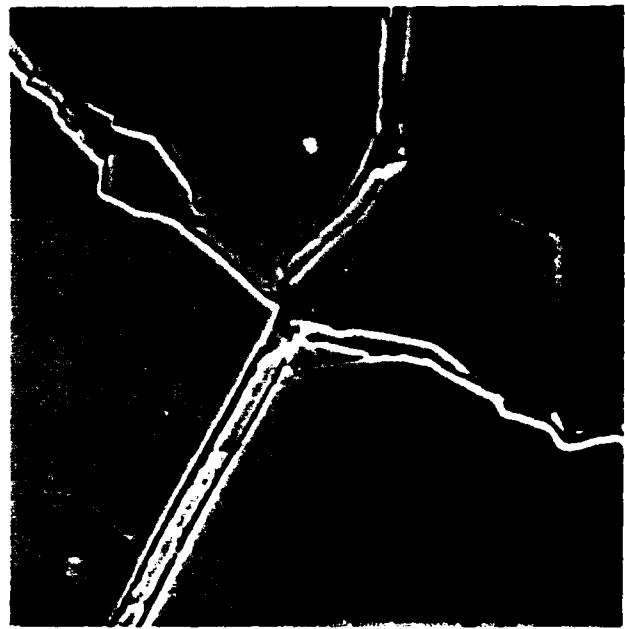
(b)

500X



(c)

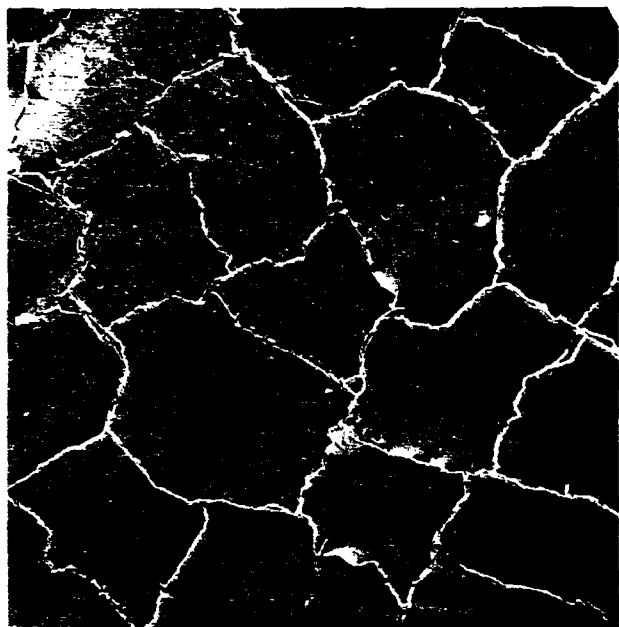
1000X



(d)

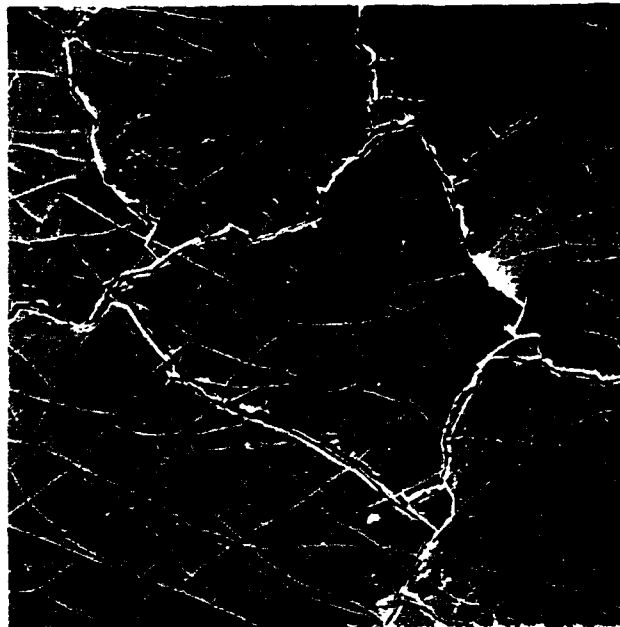
1000X

Scanning electron micrographs of mechanically polished surface of chromium on a fired cannon.



(a)

100X



(b)

200X



(c)

500X



(d)

1000X

Scanning electron micrographs of electropolished surface  
of chromium on a fired cannon.





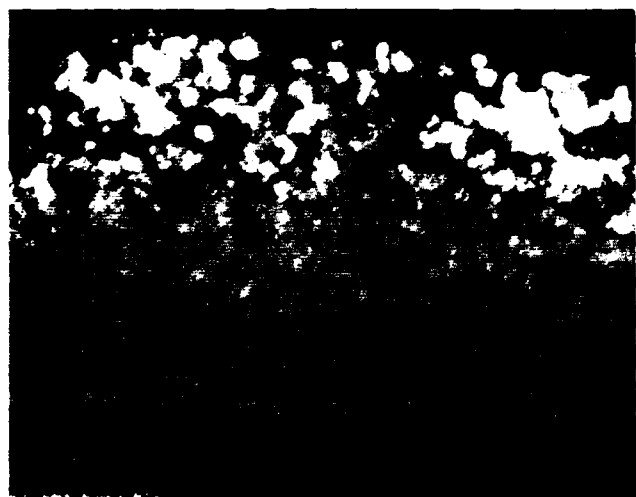
(a)

10,000X



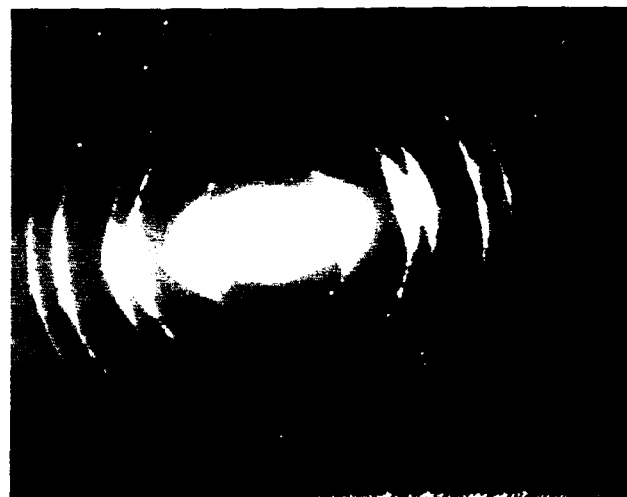
(b)

60,000X



(c)

30,000X



(d)

SAD

HVEM of chromium electroplate stripped from surface of fired cannon.

Figure 15

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